In-situ TEM Observations of Nucleation and Growth of W-nanowires on SiO₂ Substrates in an Electron-beam-induced Deposition Process

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Electron-beam-induced deposition (EBID) is one of the most promising techniques to fabricate small-sized structures on substrates. It provides a method for obtaining features of different sizes, shapes, and materials in submicron or nanometer scales [1]. Compact structures are usually fabricated on conducting substrates with this technique. Recently, by utilizing insulator substrates of Al_2O_3 , W- nanowires, nanodendrites, or nano-fractal-like trees have been fabricated with EBID using a precursor W(CO)₆ in a 200 kV transmission electron microscope (TEM) [2]. The structures were also successfully fabricated on SiO₂ substrates [3]. The development of different morphologies of these structures was very dependent on the electron irradiation condition. A well understanding of the details of nucleation and growth of the nano-structures is necessary not only for the application of this technique, but also for scientific interest for understanding the physical phenomenon. The aim of the present work is to investigate the nucleation and growth of W-nanowires on SiO₂ with an in-situ TEM observation method.

EBID was performed in a high voltage transmission electron microscope (HVTEM), JEM-ARM1000, operated at 400 kV. $W(CO)_6$ powder was used as a precursor [2]. Crystalline SiO₂ specimens suitable for observation with TEM were prepared by cutting, grinding, polishing, and Ar ion milling. The experiment was carried out at room temperature.

Fig. 1 shows a series of TEM micrographs recorded with a video system during the EBID. The electron beam intensity for irradiating the specimen was about 0.26 A cm⁻², which was estimated by measuring the total electron beam current and the beam size on the specimen. The irradiation was begun at 0 second in a clean area on the substrate as shown in fig. 1a. Tiny dot-like contrasts smaller than 2 nm were observed after 5 seconds irradiation as shown in fig. 1b. The tiny dots grew larger with the time. The dots begun to grow out of the surface of the substrate after 13 seconds as shown in fig. 1c and d. After the irradiation for 19 seconds, shapes of nanowires could be identified. At 85 second, the lengths of the nanowires were in order of about 10 nm. The nanowires continued to grow with increasing the time. However, some of the nanowires branch at tips after the irradiation for about 85 seconds as shown in fig. 1f. The similar processes of nucleation and growth of nanowires under more intensive electron beam irradiation were also observed. Fig. 2 shows the TEM micrographs of the nanowires fabricated with electron beam intensity of about 0.26 A cm⁻² (a) and 0.86 A cm⁻² (b), respectively. The irradiation period after the beginning of nucleation was 237 seconds for fig. 2a and 220 seconds for fig. 2b, respectively. The nanowires fabricated with beam intensity of 0.86 A cm⁻² presented a better evenness in length and thickness.

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Fig. 1. TEM micrographs of Nano-wires fabricated on a SiO_2 substrate with EBID in an electron beam intensity of 0.26 A cm⁻² recorded at different irradiation times. (a) to (f) at 0, 5, 13, 19, 51 and 85 seconds, respectively.



Fig. 2. TEM micrographs of Nano-wires fabricated on a SiO₂ substrate with EBID in electron beam intensities of 0.26 A cm⁻² (a) and 0.86 A cm⁻², respectively.